REPORT ON ARSENIC

By C. W. MURRAY (Eastern Regional Research Laboratory, Bureau of Agricultural Chemistry and Engineering, U. S. Department of Agriculture, Philadelphia, Pa.), *Associate Referee*

Since his appointment in the spring of the present year, the Associate Referee has been unable to do any chemical work on methods. Therefore, only the results of a collaborative study of the Cassil-Wichmann method, as modified by them, *This Journal*, **23**, 297 (1940), will be presented at this time.

RESULTS

The results obtained by the various collaborators are given in Table 1. The figures for the arsenic solutions are averages based on two or three determinations, and those for shrimp and tobacco are averages of from two to five determinations and two digestions.

TABLE 1.—*Collaborative results on three arsenic solutions and a sample each of shrimp and tobacco*

COLLABORATOR	ARSENIC SOLUTIONS NO. 1	NO. 2	NO. 3	As_2O_3 (P.P.M.) SHRIMP	TOBACCO
	GAMMA As_2O_3/10 ML.				
	18.0	92.0	487.0		
American Can Co.					
O. F. Ecklund	17.2	96.4	465.0	5.0	35.6
California Dept. Agr.					
W. G. Marshall	16.9	89.3	480.0	3.1	37.1
Food and Drug Adm.					
S. Alfend	16.8	87.9	457.0	3.7	38.8
S. D. Fine	16.7	89.7	451.0	3.6	38.5
D. A. Ballard	16.5	88.7	464.2	4.6	33.7
L. W. Ferris	17.3	90.9	469.5	4.9	42.0
P. A. Mills	17.0	90.2	471.4	4.0	33.9 (2d wet ash); 39.8 (3d wet ash); 30.1 (4th wet ash)
A. K. Klein	17.6	88.9	477.0	4.5	33.3
D. M. Taylor	18.3	92.9	487.5	4.5	41.4
Bur. Ent. & Plant Quar.					
L. Koblitsky	19.5	99.6	449.0	7.1	40.4
J. E. Fahey	17.4	90.5	474.6	3.9	39.7
J. F. Cassidy	18.5	89.7	450.0	4.7	34.7
C. C. Cassil	19.6	92.3	480.0	6.6	39.6
J. H. Jones	18.7	88.4	453.0	—	—
Average	17.7	91.1	466.4	4.6	37.6
Av. % recovered	98.3	99.0	95.8	—	—
Range of recovery Percentages	91.7–108.9	95.5–108.3	92.2–100.1	—	—

The comments of the various collaborators may be summarized as follows:

(1) The evolution of arsine is accompanied by too much foaming even after the addition of lead acetate.

(2) Several investigators had difficulty in dissolving the brownish-yellow deposit on the leucite tube.

(3) The solution can not be brought to boiling in 2 minutes because of the rapid evolution of hydrogen. The boiling point is reached only near the end of the evolution.

(4) In preliminary runs prior to actual analysis of the samples, several investigators report low recoveries—some as low as 92.5%.

(5) Several investigators are not satisfied with their results for Sample 3, and they suggest that this may be the limit of the method.

(6) Some doubt as to the accuracy of the shrimp analyses is raised.

(7) It is pointed out that by actual test all the arsine is not evolved in 7 minutes.

RECOMMENDATIONS*

It is recommended—

(1) That the text be revised to permit longer arsine evolution, perhaps 10 minutes; to bring the solution to a boil more slowly (5 or 6 minutes); and to provide that the precipitate on the leucite tube be given a minute or two to dissolve.

(2) That the upper limits claimed for the method be restudied as well as the analysis for arsenic in shrimp.

(3) That since the results of the tobacco analyses were only fair this study be repeated and that a peroxide combustion be tested as a possible replacement operation for the usual wet digestion now used for shrimp and tobacco.

REPORT ON COPPER

By C. A. GREENLEAF (National Canners Association, Washington, D. C.), *Associate Referee*

The reports on copper during the past several years have established the merits of the colorimetric method with sodium diethyldithiocarbamate as a reagent and have demonstrated most of the necessary conditions for its successful use. The principal task this year was to consolidate this information in explicit form. In addition, further study was given to methods for overcoming the interference of bismuth, cobalt, and nickel.

In the latter phase of the work attention was given to the hydrogen sulfide separation and to a preliminary extraction with dithizone. The hydrogen sulfide separation was included in the collaborative work reported by Coulson, *This Journal*, 19, 219 (1936), who discussed the merits of this method for separation of small quantities of copper. Apparently

* For report of Subcommittee C and action by the Association, see *This Journal*, 24, 58 (1941).